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Accelerated soak performance of BPDA-PPD polyimide for implantable MEAs

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Abstract

To mature toward real-world applications biomedical sensors should maintain long-term functionality under relatively harsh in-vivo conditions. Microelectrode arrays (MEAs) rely on thin films, such as parylene C or polyimide, the water-barrier of which is crucial for the long-term stability. This work reports on the high water-barrier of BPDA-PPD polyimide. This material was spin-coated on interdigitated microstructures and immersed in saline@60°C for 170 days. The interdigitated impedance remained stable, higher than parylene C. Curing was crucial for the water-barrier of this polyimide: only long-cured microelectrode arrays remained unchanged in their inter-electrode impedances as compared to short-cured devices when soaked in saline@60°C for 140 days. Subjecting BPDA-PPD polyimide foils to water-vapour penetration revealed higher performance than parylene C. These results disclose BPDA-PPD polyimide as a promising insulation for implantable MEAs.

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Keywords: BPDA-PPD polyimide, U-Varnish, biomedical, ECoG, parylene C, encapsulation, accelartion soak test, aging, water-barrier, MEA

1. Motivation

Flexible biomedical devices have recently emerged to enable novel methods of signal-acquisition from within the human body. In this regard, ECoG MEAs – only several microns thick – can acquire electrical activity of neuronal populations directly from the cortical surface [1]. Herewith they offer information about cognitive behaviour of high speed and accuracy for future weakly-invasive BCIs and neuroprosthetics [2]. Nevertheless, exactly the highly

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miniaturized nature of ECoG MEAs makes them highly susceptible to diffusion of salt water – most abundant medium in-vivo – and herewith to the formation of parasitic short-circuits between the electrode traces of the MEAs.

A prerequisite for ECoG devices to enter the market is to guarantee their in-vivo functionality for a span of several years. Established packaging methods for established medical devices such as heart-pace makers and cochlear implants cannot be applied because of the highly miniaturized nature of ECoG MEAs. Instead, polymeric materials, such as parylene C or polyimide have served as thin-film isolation in most ECoG devices. The long-term in-vitro stability of parylene C has been thoroughly investigated [3]. The BPDA-PPD polyimide has lowest moisture absorption among the polyimides and was shown to maintain its mechanical properties in-vitro [4].

We used this polyimide for the fabrication of an ECoG MEA [1] and present here the in-vitro performance of a 5µm-thick BPDA-PPD polyimide fabricated as: (1) a thin-film coating on interdigitated electrodes and (2) flexible insulation of an ECoG MEA; (3) thin-foil specimen subjected to water-vapour penetration.

2. Materials and Methods

2.1. Microfabrication

Three samples sets were fabricated: 1). interdigitated electrodes (IDE) coated with short-cured BPDA-PPD polyimide, long-cured one and parylene C as a reference; 2). MEAs made from short-cured BPDA-PPD polyimide and long-cured one; 3). thin foils made from long-cured BPDA-PPD polyimide.

1. IDE samples (see Fig.1): A borosilicate glass wafer was cleaned in Caro's acid and sputter-coated with a 20/300nm thick Cr/Au. The Au surface was treated in O_2 plasma (500W, 30s) to promote good adhesion and a 1.8µm-thick AZ1518 photoresist was spin-coated, exposed for 2.5s@ 12mW/cm^2 and developed in AZ 726 MIF. The photoresist structure was transferred into Au by wet etching it in a Cyanide etch solution. The resist was stripped in acetone & IPA, and the Au structure was transferred into Cr by wet etching. The wafer was cleaned in Caro's acid and coated by either a 5µm-thick parylene C or a U-Varnish-S polyimide film (BPDA-PPD PI), whereas the polyimide film was either short or long-cured (see Fig.2). A 10µm-thick AZ 9260 photoresist was spin coated and structured into contact openings and the parylene/polyimide was etched in an O_2+CF_4 RIE-plasma. The resist was stripped in AZ100 (15min@50°C). The wafer was diced.

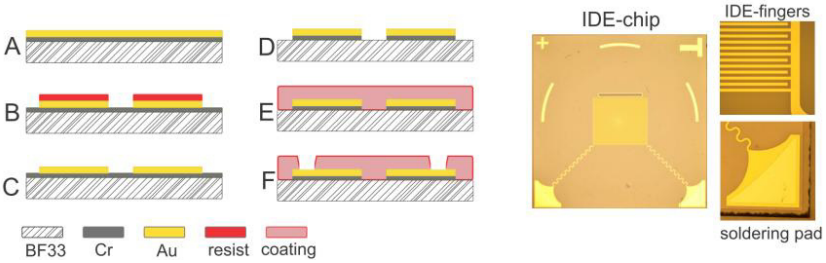

Fig. 1. Microfabrication flow of an IDE sensor with dimensions: 15mm x 15mm IDE-chip and 15µm-wide IDE fingers.

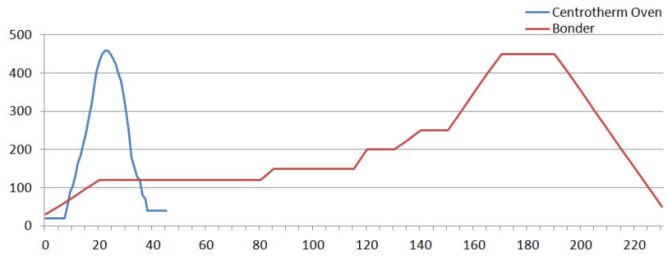

Fig. 2. Curing profile of polyimide coating in a Centrotherm oven (short cure in N_2) and a wafer-bonding tool (long cure in vacuum ambient).

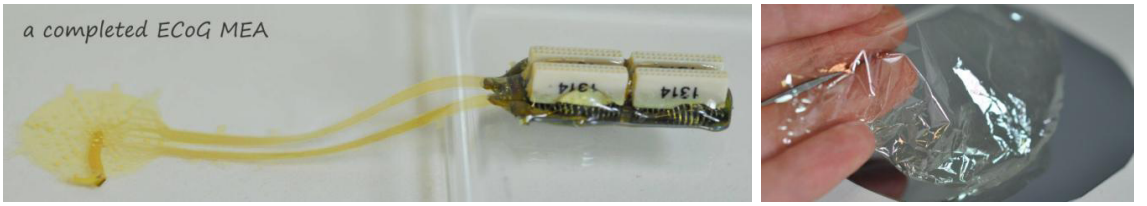

Fig. 3. **Left image:** BPDA-PPD polyimide-insulated ECoG MEA; **Right image:** 5µm-thick BPDA-PPD polyimide foil peeled off the wafer.

2. MEA samples (Fig.3, left) were fabricated as described in [3].

3. Foil samples (Fig.3, right): 4" Si wafers were cleaned in Caro's acid and their native oxides were stripped with a HF dip. Straight afterwards the organosilane adhesion promoter VM651 was spin-coated for 60s@4000rpm and backed on a hotplate for 120s@120 ◦ C. A 5µm-thick U-Varnish-S film (BPDA-PPD) was spin-coated for 40s@3000rpm and soft-backed for 5min@80-120 ◦ C & 5min@120 ◦ C on a hotplate. The samples were cured in a Substratbonder SUESS SB6 (long-curing) in a vacuum ambient; (2) chamber evacuation. After the curing, the wafers were kept for a week in a wafer box. The polyimide films were then peeled off the wafers as follows: (1) a razor was used to remove the PI residues from the wafer edges, and (2) a rounded piece of paper to stepwise detach the polyimide foil off its wafer.

2.2. Measurement Test Set-Up

1. IDE samples (Fig.4): glass cylinders were fixed on top, filled with saline (Fig.1) and kept @60°C for 170 days. If water was absorbed by the coating, the impedance was reduced, which was monitored by an electrochemical impedance spectroscope (EIS).
2. MEA samples (Fig.5): two MEAs were fabricated from BPDA-PPD PI: a short-cured and a long-cured one. The electrode openings of the MEAs (circular portion in Fig.3, left) were kept in saline @60°C for 140 days. The impedances between electrode pairs (inter-electrode impedances) were monitored with a multiplexer linked to an EIS, whereas an impedance decline was associated with water diffusion – absorption by the polyimide and/or lateral creeping along interfaces.
3. Foil samples: long-cured BPDA-PPD foils were subjected to water-vapour permeation analysis (two samples). The water-vapour permeation tests were performed externally at PAUL LIPPKE HANDELS-GMBH (www.mocon.eu) at 23° C, 85%RH.

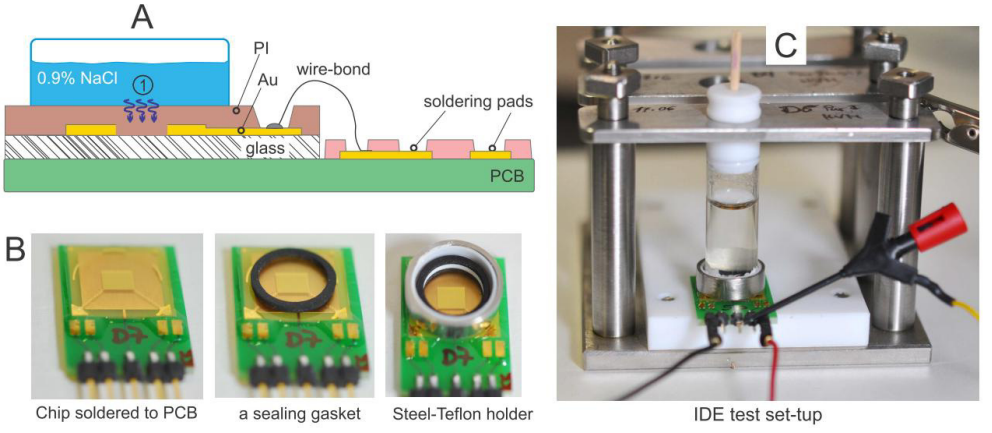

Fig. 4. IDE-soak test: (A) schematic of sample cross-section; (B) Assembly of sealing gaskets on IDE-chips; (C) measurement set-up.

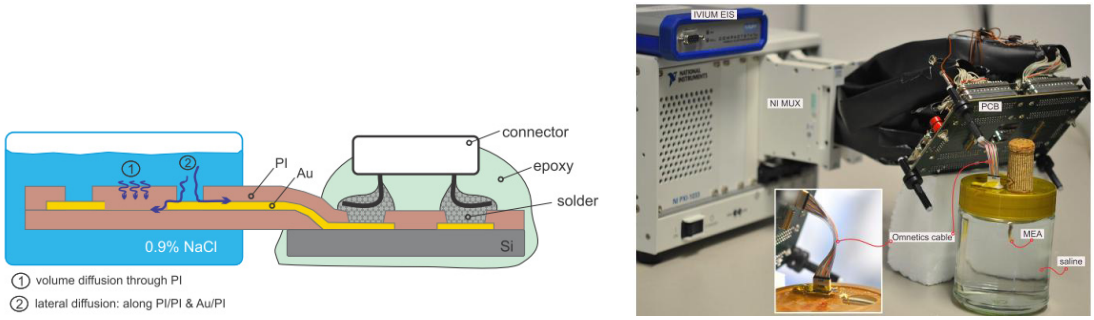

Fig. 5. **Left image:** A flex-rigid MEA partially submerged in saline; **Right image:** The MEA is linked to a PCB to a NI multiplexer and an IVIUM impedance meter.

3. Results

3.1. IDE samples

Fig.6 shows the impedance data obtained from the IDE samples: short-cured PI, parylene C and long-cured PI. Polyimide curing is essential for the establishment of a high water-barrier of BPDA-PPD polyimide: short-cured PI films decreased early in their impedance values (Fig.6, left); parylene C was stable with a slight decline in impedance (Fig.6, middle), whereas long-cured BPDA-PPD PI remained most stable (Fig.6, right), even superior to Parylene C.

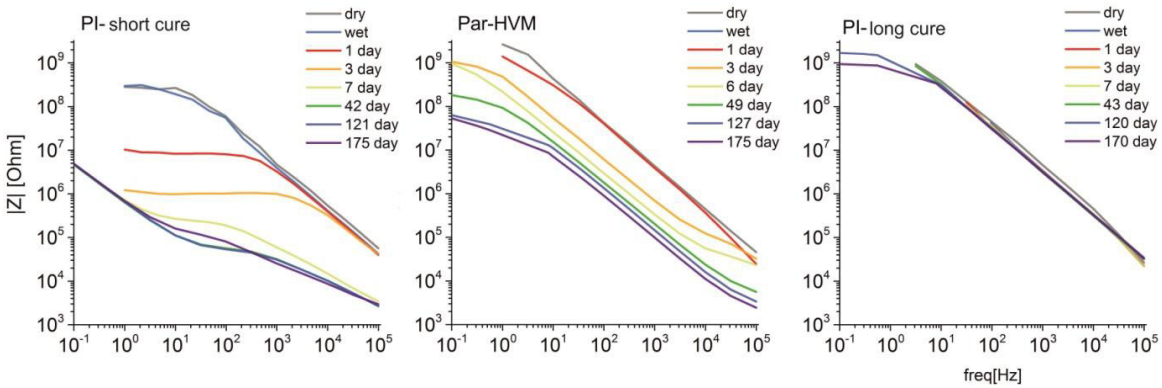

Fig. 6. Results from in-vitro IDE-tests: short-cured BPDA-PPD polyimide declined quickly in impedance values (left image); Parylene C with Silane A-174 adhesion promoter remained relatively high impedance values, with a slight decline (middle image); Long-cured BPDA-PPD polyimide remained with most stable impedance values (right image); min. 3 samples per each polyimide type and 2 samples per parylene C.

3.2. MEA samples

Short-cured MEA declined in its inter-electrode impedance (see Fig.7, left image), whereas the long-cured MEA maintained its inter-electrode impedance values (see Fig.7, right image).

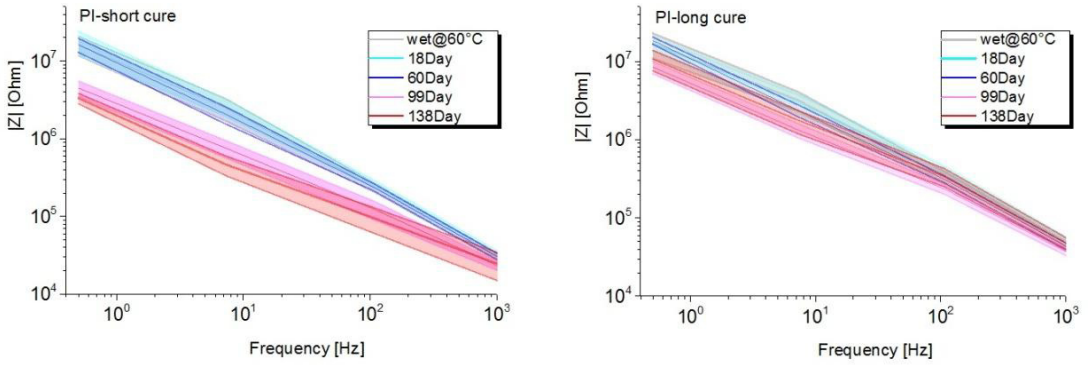

Fig. 7. Results from MEA soak tests: short-cured BDDA-PPD PI samples declined in inter-impedance values because of parasitic short-circuits formation due to saline diffusion (left image); long-cured PI MEAs remained stable, with a slight decline in inter-electrode impedances.

3.3. Foil samples

The water-vapour permeation of BPDA-PPD PI foils was measured to be 0.022 g•mm/m2•day at 23°C, 85%RH, i.e. considerably lower than that of parylene C (0.083 g•mm/m2•day at 38°C, 90%RH [5]).

4. Conclusion

We discovered that BPDA-PPD polyimide – provided it is pre-cured sufficiently long time (long-cured PI samples) – maintains an exceptionally high lateral impedance magnitude measured by IDE-structures, even higher than that of parylene C. Furthermore, MEA samples encapsulated in long-cured BPDA-PPD polyimide remained stable in their impedance values despite the fact that saline could enter the device via the electrode openings. Letting water-vapour permit the long-cured BPDA-PPD PI foils revealed an exceptionally high water-vapour barrier, even higher than that of parylene C. A possible explanation for the excellent water-barrier properties of this polyimide is that it is build from polymer chains arranged in a densely-packed “poly-crystalline” blocks and the water molecules have difficulty to trespass it on their diffusion via the polyimide film.

Acknowledgements

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